OMG AG & Co. KG

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## Claims

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Process for preparing a Palladium(0) compound, comprising reaction of a palladium compound with one or more compounds of the general formula I in the presence of a base:

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$$R^1$$
 $(A)_x$ 
 $R^4$ 
 $(I)$ 
 $R^2$ 
 $R^3$ 
 $R^6$ 
 $R^5$ 

in which:

each A is independently a CR<sup>7</sup>R<sup>8</sup>-radical where one of the A radicals may be oxygen, sulphur, an NR<sup>9</sup> group or an SiR<sup>10</sup>R<sup>11</sup> group, or where the A radicals may be a constituent of a 5- to 20-membered ring system,

x is an integer from 2 to 4, and

each  $R^1$  to  $R^{11}$  is independently selected from R, OR, halogen, CN,  $NO_2$ ,  $NR_2$ , C(O)R, C(O)OR, OC(O)R, 20 CONR<sub>2</sub>, NHCO<sub>2</sub>R, NHCOR, CH=CH-CO<sub>2</sub>R, Si(R)<sub>3</sub>, Si(OR)<sub>3</sub>,  $SiR(OR)_2$ ,  $SiR_2(OR)$ ,  $SO_3R$ ,  $SO_2R$ , SOR, SR,  $PR_2$ ,  $POR_2$ , PO<sub>3</sub>H, PO(OR)<sub>2</sub>, in which R is a hydrogen atom, a substituted or unsubstituted  $C_{1-10}$ -alkyl radical, a unsubstituted, mono-25 substituted or polyunsaturated  $C_{1-10}$ -alkenyl radical, unsubstituted, optionally substituted or heteroatom-containing  $C_{5-10}$ -aryl radical, and the

substituents on the alkyl radical or the alkenyl

radical are selected from halogen,  $O-C_{1-10}-alkyl$ , phenyl, O-phenyl, OH,  $NH_2$  and halogenated  $C_{1-10}-alkyl$ , and the substituents on the aryl radical are selected from halogen,  $C_{1-10}-alkyl$ ,  $O-C_{1-10}-alkyl$ , phenyl, O-phenyl, OH,  $NH_2$  and halogenated  $C_{1-10}-alkyl$ , where  $R^2$  and  $R^3$  and/or  $R^5$  and  $R^6$  together with the carbon atoms bonded thereto may be a constituent of a 5- to 7-membered, optionally heteroatom-containing ring.

10 2. Process according to Claim 1, wherein x is 3.

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- 3. Process according to either of the preceding claims, wherein  $\mathbb{R}^1$  to  $\mathbb{R}^6$  are each hydrogen atoms.
- 4. Process according to any of the preceding claims, wherein  $-(A)_x$  is a group of the formula  $-CH_2-X-CH_2- \text{ and } -X- \text{ is selected from } -O-, -S-, \\ -SiR_2-, -NR- \text{ and } -NC(O)R, \text{ and } R \text{ is hydrogen, a } C_{1-4}-alkyl \text{ radical or a halogenated } C_{1-4}-alkyl.$
- 5. Process according to Claim 1, wherein the compound of the general formula I is selected from 1,5-hexadiene, 1,6-heptadiene and 1,7-octadiene.
- Process according to Claim 1, wherein the compound 6. of the general formula I is selected from diallyl diallylamine, diallylmethylamine, ether, diallylethylamine, N-acetyldiallylamine, diallyl diallylsilane, diallyldimethylsilane, 25 sulphide, difurfuryl ether, difurfurylamine, bis(thiophen-2ylmethyl)amine, difurfuryl sulphide and 1,3divinylbenzene.
- 7. Process for preparing a Palladium(0) compound,
  30 comprising reaction of a palladium compound with

one or more compounds of the general formula II in the presence of a base:

in which:

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5 n is an integer from 3 to 20,

each  $R^{13}$  to  $R^{15}$  is independently selected from R, OR, halogen, CN,  $NO_2$ ,  $NR_2$ , C(O)R, C(O)OR, OC(O)R,  $CONR_2$ ,  $NHCO_2R$ , NHCOR,  $CH=CH-CO_2R$ ,  $Si(R)_3$ ,  $Si(OR)_3$ ,  $SiR(OR)_2$ ,  $SiR_2(OR)$ ,  $SO_3R$ ,  $SO_2R$ , SOR, SR,  $PR_2$ ,  $POR_2$ , which R is hydrogen,  $PO_3H$ , PO(OR)<sub>2</sub>, in substituted or unsubstituted  $C_{1-10}$ -alkyl radical, a substituted or unsubstituted, mono- $C_{1-10}$ -alkenyl radical, polyunsaturated unsubstituted, substituted or optionally heteroatom-containing  $C_{5-10}$ -aryl radical, and the substituents on the alkyl radical or the alkenyl radical are selected from halogen, O-C<sub>1-10</sub>-alkyl, phenyl, O-phenyl, OH,  $NH_2$  and halogenated  $C_{1-10}$ alkyl, and the substituents on the aryl radical are selected from halogen,  $C_{1-10}$ -alkyl,  $O-C_{1-10}$ alkyl, phenyl, O-phenyl, OH,  $\mathrm{NH}_2$  and halogenated  $C_{1-10}$ -alkyl, where  $R^{13}$  and  $R^{14}$  together with the carbon atoms bonded thereto may be a constituent of a 5- to 7-membered, optionally heteroatomcontaining ring, and

each  $R^{12}$  is independently selected from hydrogen, a hydroxyl group, a substituted or unsubstituted  $C_{1-10}$ -alkyl radical, an  $-O-C_{1-10}$ -alkyl radical (where

the alkyl radical may be substituted or unsubstituted), a substituted or unsubstituted, mono- or polyunsaturated  $C_{1-10}$ -alkenyl radical or a substituted or unsubstituted, optionally heteroatom-containing  $C_{5-10}$ -aryl radical, where the substituents are as defined for  $R^{13}$  and  $R^{15}$ .

8. Process according to Claim 7, wherein n is an integer from 3 to 6 and each  $R^{12}$  is independently a  $C_{1-4}$ -alkyl radical or a halogenated  $C_{1-4}$ -alkyl radical.

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- 9. Process for preparing a Palladium(0) compound, comprising reaction of a palladium compound with one or more compounds of the general formula III in the presence of a base:

v and w are each independently 0 or an integer of from 1 to 1000 and v+w is from 0 to 1000,

each R16 is independently selected from hydrogen, a hydroxyl group, a substituted or unsubstituted 20  $C_{1-10}$ -alkyl radical, an  $-O-C_{1-10}$ -alkyl radical (where the alkyl radical may be substituted unsubstituted), a substituted or unsubstituted, mono- or polyunsaturated  $C_{1-10}$ -alkenyl radical or a 25 substituted unsubstituted, optionally heteroatom-containing  $C_{5-10}$ -aryl radical, where the substituents are as defined for  $R^{17}$  and  $R^{19}$ ,

each  $R^{17}$  to  $R^{19}$  is independently selected from R, OR, halogen, CN, NO<sub>2</sub>, NR<sub>2</sub>, C(0)R, C(0)OR, OC(0)R,

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CONR<sub>2</sub>, NHCO<sub>2</sub>R, NHCOR, CH=CH-CO<sub>2</sub>R, Si(R)<sub>3</sub>, Si(OR)<sub>3</sub>,  $SiR(OR)_2$ ,  $SiR_2(OR)$ ,  $SO_3R$ ,  $SO_2R$ , SOR, SR,  $PR_2$ ,  $POR_2$ , PO<sub>3</sub>H, PO(OR)<sub>2</sub>, in which R is a hydrogen atom, a substituted or unsubstituted  $C_{1-10}$ -alkyl radical, a substituted or unsubstituted, monopolyunsaturated  $C_{1-10}$ -alkenyl radical, substituted or unsubstituted, optionally heteroatom-containing  $C_{5-10}$ -aryl radical, and the substituents on the alkyl radical or the alkenyl radical are selected from halogen, O-C<sub>1-10</sub>-alkyl, phenyl, O-phenyl, OH,  $NH_2$  and halogenated  $C_{1-10}$ alkyl, and the substituents on the aryl radical are selected from halogen,  $C_{1-10}$ -alkyl,  $O-C_{1-10}$ alkyl, phenyl, O-phenyl, OH, NH2 and halogenated  $C_{1-10}$ -alkyl, where  $R^{17}$  and  $R^{19}$  together with the carbon atoms bonded thereto may be a constituent of a 5- to 7-membered, optionally heteroatomcontaining ring,

each  $R^{20}$  is independently selected from hydrogen, a hydroxyl group, a substituted or unsubstituted  $C_{1-10}$ -alkyl radical, an  $-O-C_{1-10}$ -alkyl radical (where the alkyl radical may be substituted or unsubstituted), a substituted or unsubstituted, mono- or polyunsaturated  $C_{1-10}$ -alkenyl radical, or a substituted or unsubstituted, optionally heteroatom-containing  $C_{5-10}$ -aryl radical, where the substituents are as defined for  $R^{17}$  and  $R^{19}$ , and

each Term is independently  $(R^{16})_2(CR^{17}R^{18}CR^{19})$  Si- or  $(R^{16})_3$ Si-.

30 10. Process according to Claim 9, wherein the compound of the general formula (III) has the general formula:

## Term-O-[Si( $R^{16}$ )( $CR^{19}CR^{17}R^{18}$ )O]<sub>v</sub>-Term

where  $R^{16}$  to  $R^{19}$ , Term and v are each as defined in Claim 9.

- 11. Process according to any of Claims 7 to 10,

  wherein the compound of the general formula II or

  III is selected from divinyldisiloxane, 1,1,3,3
  tetramethyl-1,3-divinyldisiloxane, 1,1,3,3-tetra
  methyl-1,3-dithien-2-yldisiloxane, 1,1,3,3-tetra
  methoxy-1,3-divinyldisiloxane, 1,3-dimethyl-1,3
  divinyldisiloxanediol, 1,3,5,7-tetravinyl-1,3,5,7
  tetramethylcyclotetrasiloxane and 1,3,5-trimethyl
  1,3,5-trivinylcyclotrisiloxane.
- 12. Process according to any of Claims 7 to 10, wherein the compound of the general formula II or III is selected from 1,1,3,3-tetramethyl-1,3-divinyldisiloxane, 1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetrasiloxane and 1,3,5-trimethyl-1,3,5-trivinylcyclotrisiloxane.
- 13. Process according to any of the preceding claims, wherein the palladium compound is selected from  $PdX_2$ ,  $PdX_4$ ,  $M_2PdX_4$ ,  $M_2PdX_6$ ,  $(NH_3)_2PdX_2$  and  $[Pd(NH_3)_4]X_2$ , where M is a hydrogen atom, an alkali metal or  $NR^*_4$  ( $R^*$  = hydrogen,  $C_{1-4}$ -alkyl) and X is a halogen or  $NO_3$ .
- 25 14. Process according to Claim 13, wherein X is chlorine.
  - 15. Process according to any of the preceding claims, wherein the reaction is effected in the presence of a solvent or solvent mixture.

- 16. Process according to Claim 15, wherein the solvent is selected from water,  $C_{1-6}$ -alcohols and  $C_{2-6}$ -ethers and mixtures thereof.
- 17. Process according to any of the preceding claims, wherein the base is selected from alkali metal salts, alkaline earth metal salts and ammonium salts (ammonium as  $NR_4^+$  where R = H or  $C_{1-4}$ -alkyl) of carbonates, hydrogencarbonates and hydroxides.
- 18. Process according to any of the preceding claims,10 also comprising a purification step.
  - 19. Process according to any of the preceding claims, also comprising a concentration step.
  - 20. Process according to any of the preceding claims, wherein the reaction of the palladium compound with one or more compounds of the general formula I, II or III is carried out in the presence of one or more ligands other than the compound of the general formula I, II or III.

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- 21. Process according to any of the preceding claims,
  20 further comprising the reaction of the palladium
  compound with one or more ligands other than the
  compound of the general formula I, II or III.
  - 22. Palladium(0) compound obtainable by a process according to Claim 1, wherein the compound of the general formula I is hexadiene or octadiene.
    - 23. Palladium(0) compound obtainable by a process according to Claim 9, wherein the compound of the general formula III is 1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetrasiloxane.